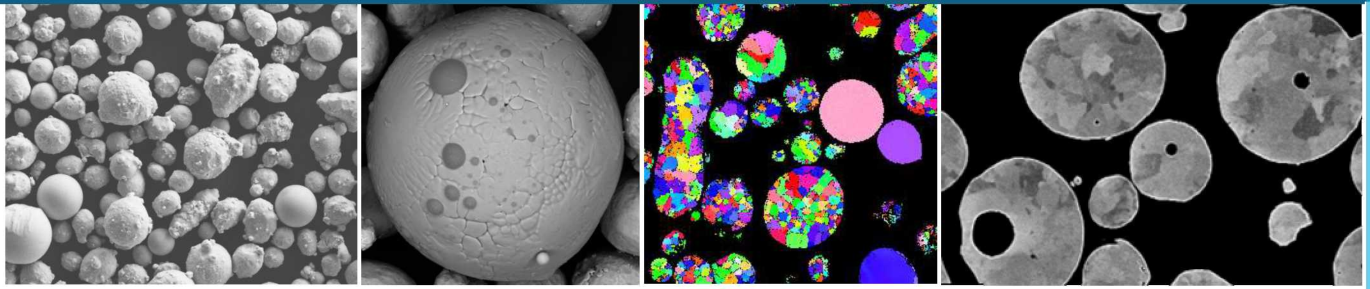
SAND2019-14399C

Pairing 316L AM Powder Feedstock with Mechanical Performance: What Really Matters



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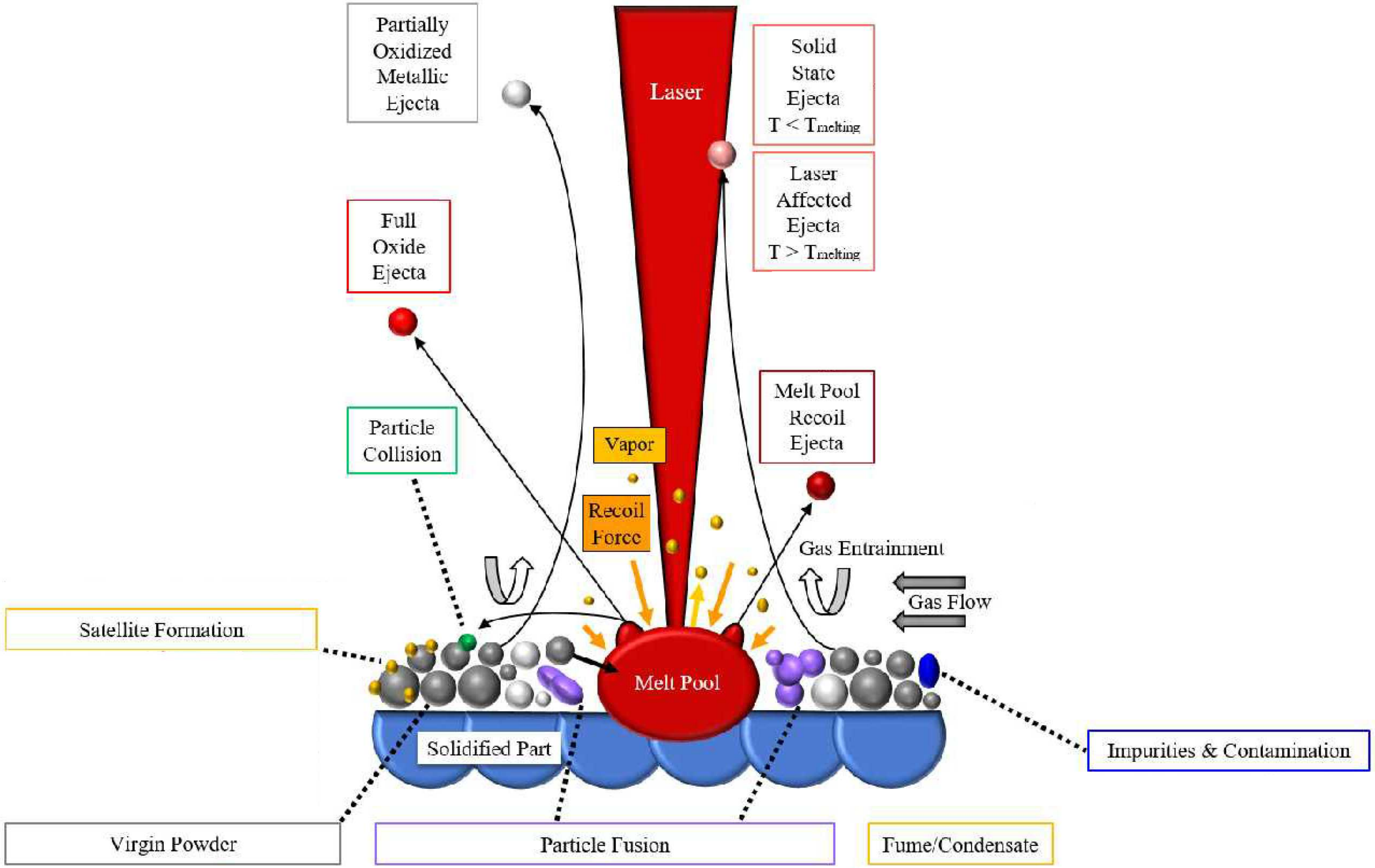
Outline

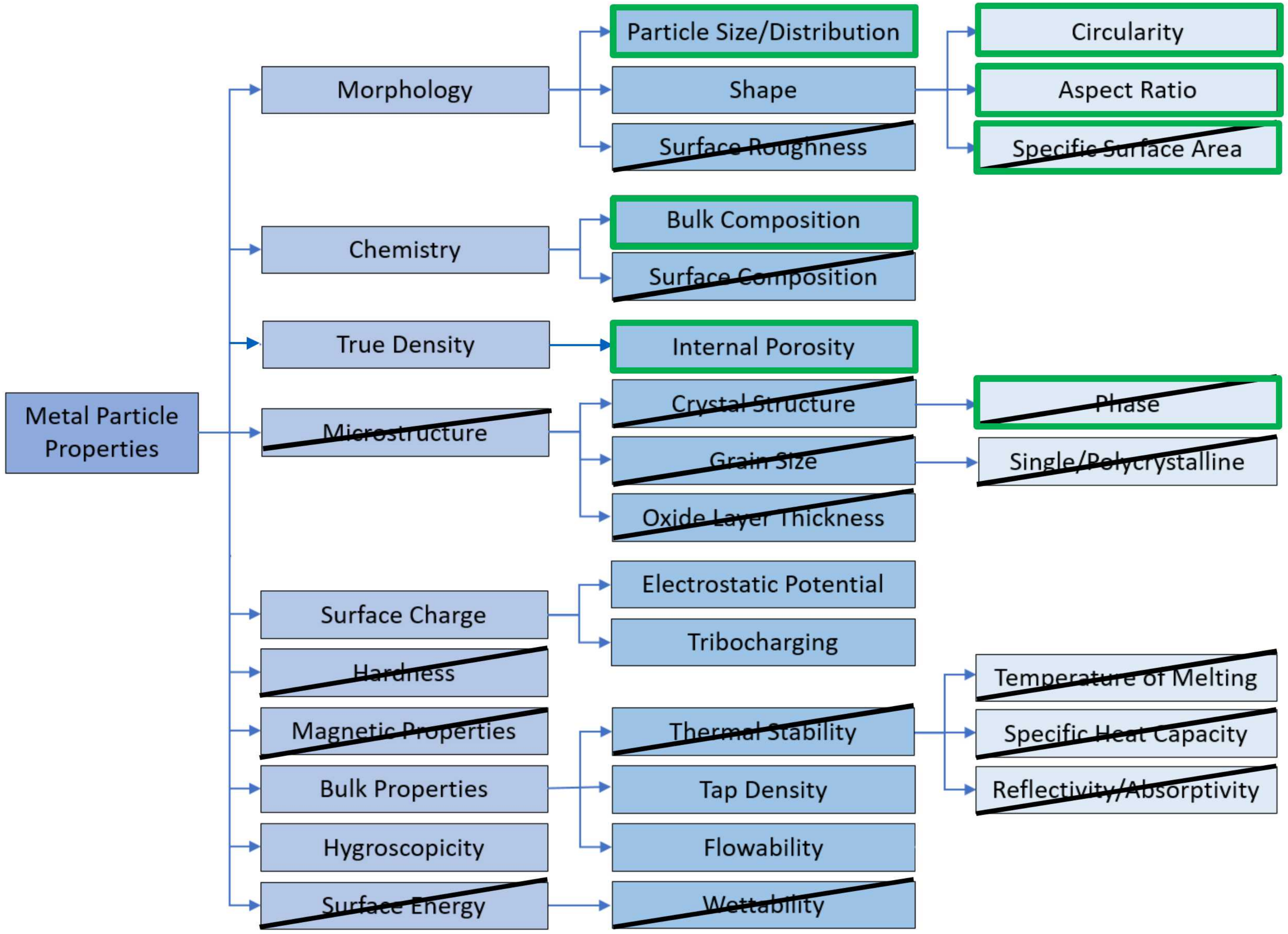
- Laser-powder interactions for 316L in L-PBF
- Powder Characteristics – Boiling Down What Matters
- Coupling Powder’s Influence on Part Mechanical Properties
  - Powder Reuse Effects on 316L Parts for Varying Lots
  - Comparing Different Initial Powders
- Takeaways for Designing an AM Powder Specification

Laser-powder interactions for 316L in L-PBF



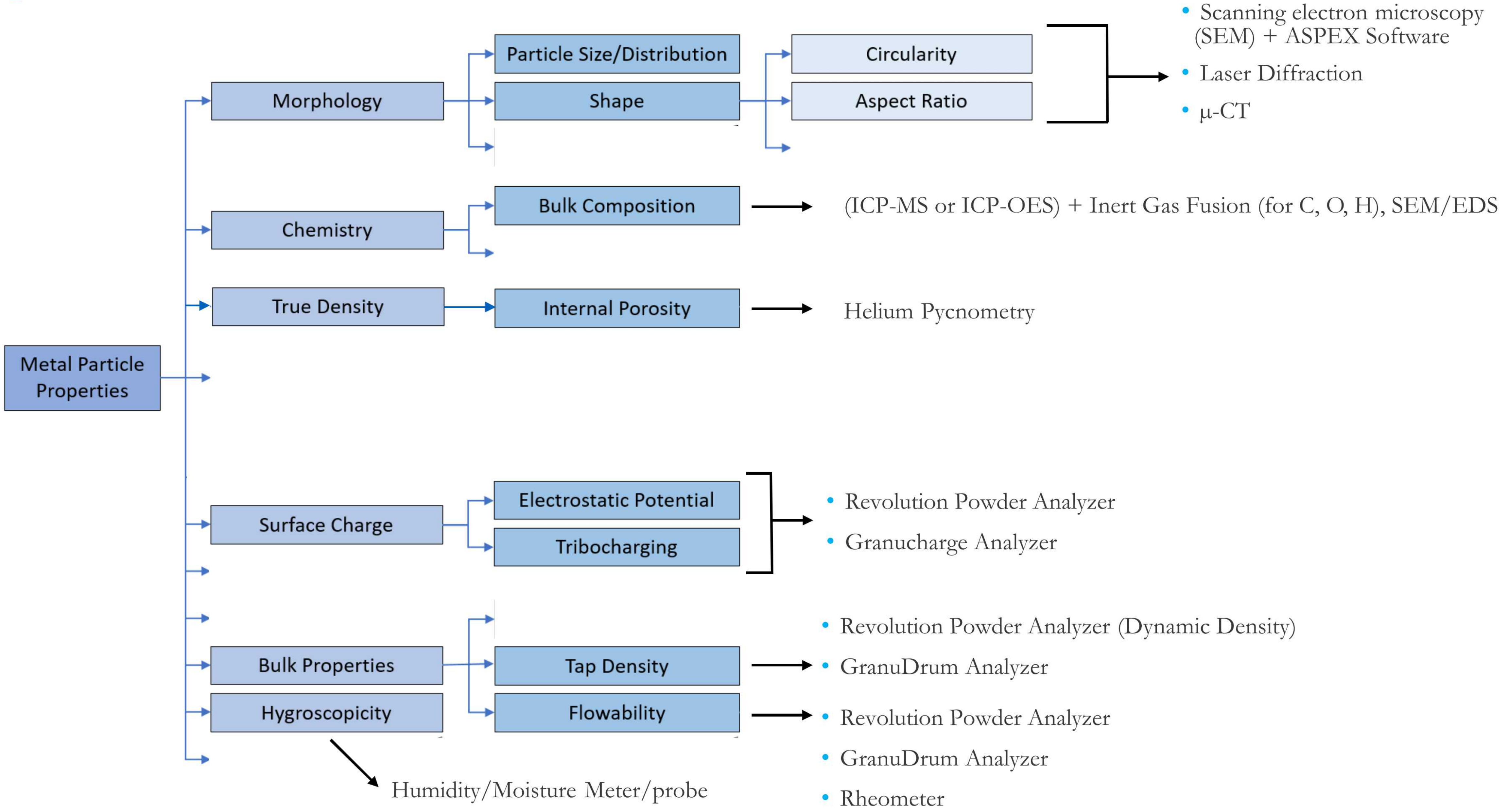
[1] Heiden, M. J., et al. (2019). "Evolution of 316L stainless steel feedstock due to laser powder bed fusion process." Additive Manufacturing **25**: 84-103.

316L Powder Characteristics – What Matters?



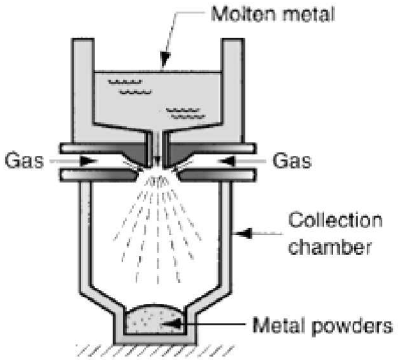
Properties crossed out are either too time intensive/expensive, shown to have no effect on part, or doesn't accurately represent whole powder lot

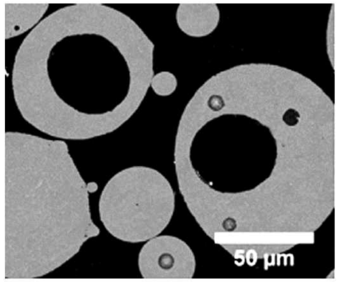
316L Powder Characteristics – What Matters?



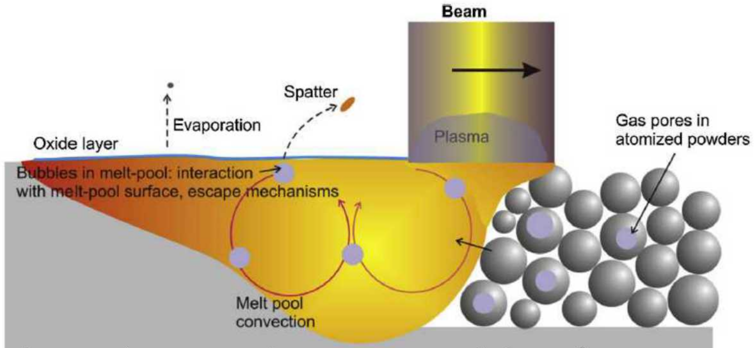
Important Powder Characteristics to Track

Internal porosity created from atomization process [1]





Porosity in gas atomized powder particles [2]

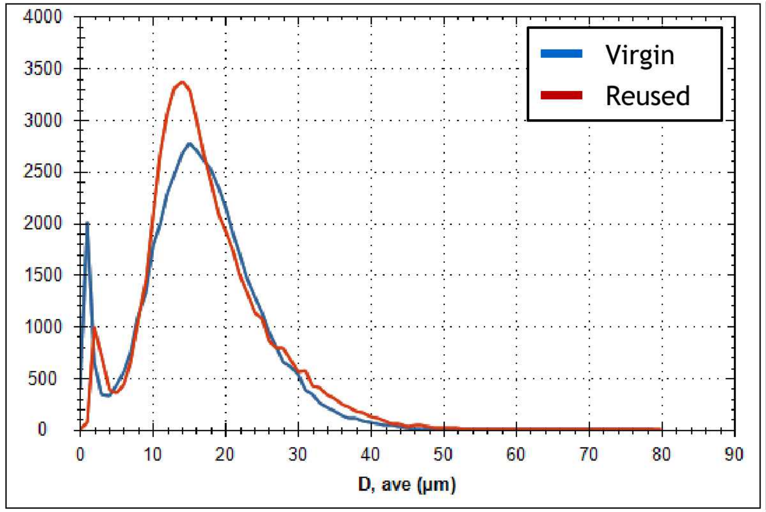


Internal gas porosity can create defects [2]

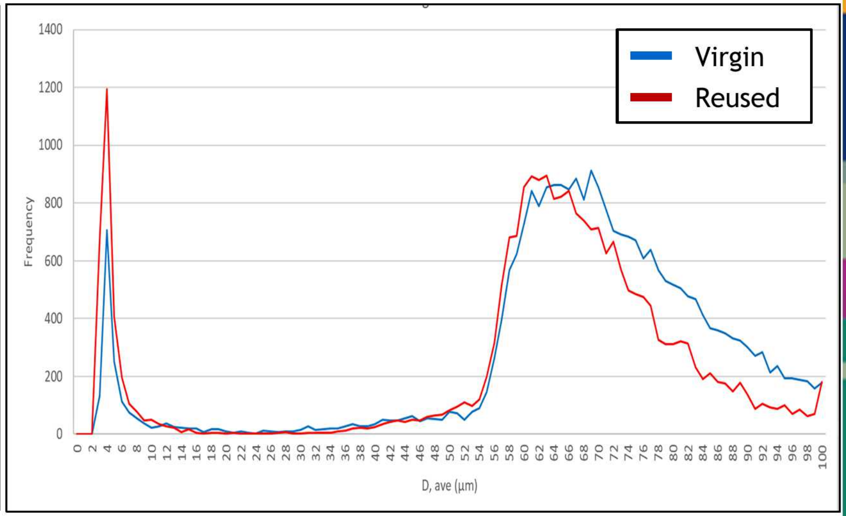
316L "bulk" chemistry changes depending on severity of machine parameters and build volume

Wt.% (Stdev)	Fe	Cr	Ni	Mo	Si	Mn	Cu	P	Co	C	S	O	N
Virgin	67.8 (0.3)	16.84 (0.34)	10.81 (0.22)	2.05 (0.20)	0.65 (0.10)	1.20 (0.12)	0.21 (0.03)	0.015 (0.002)	0.098 (0.015)	0.011 (0.002)	0.014 (0.002)	0.067 (0.010)	0.086 (0.013)
Reused (30 cycles)	67.6 (0.3)	16.91 (0.34)	10.90 (0.22)	2.02 (0.20)	0.60 (0.09)	1.27 (0.13)	0.22 (0.03)	0.016 (0.002)	0.11 (0.016)	0.016 (0.002)	0.014 (0.002)	0.095 (0.014)	0.090 (0.013)
ASTM Spec [2]	61-69	16-18	10-14	2-3	1 max	2 max	-	0.045 max	-	0.03 max	0.03 max	-	-

L-PBF reuse coarsens particles [3]



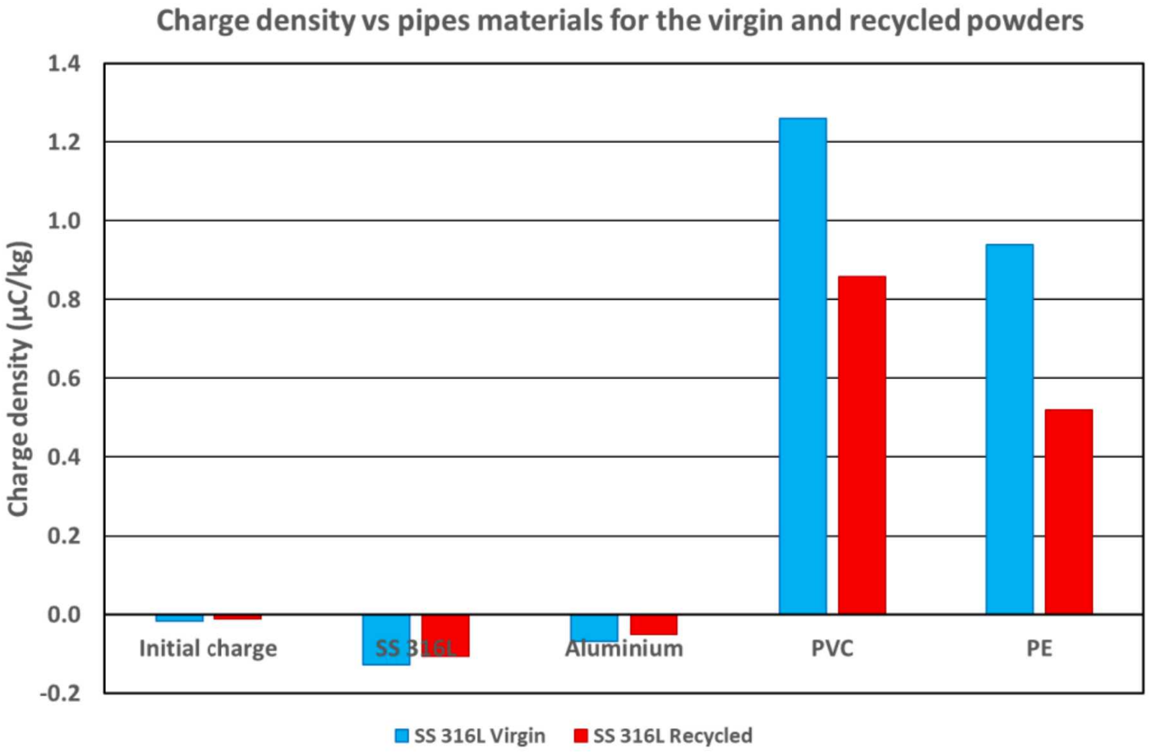
DED (LENS) reuse reduces coarse particles



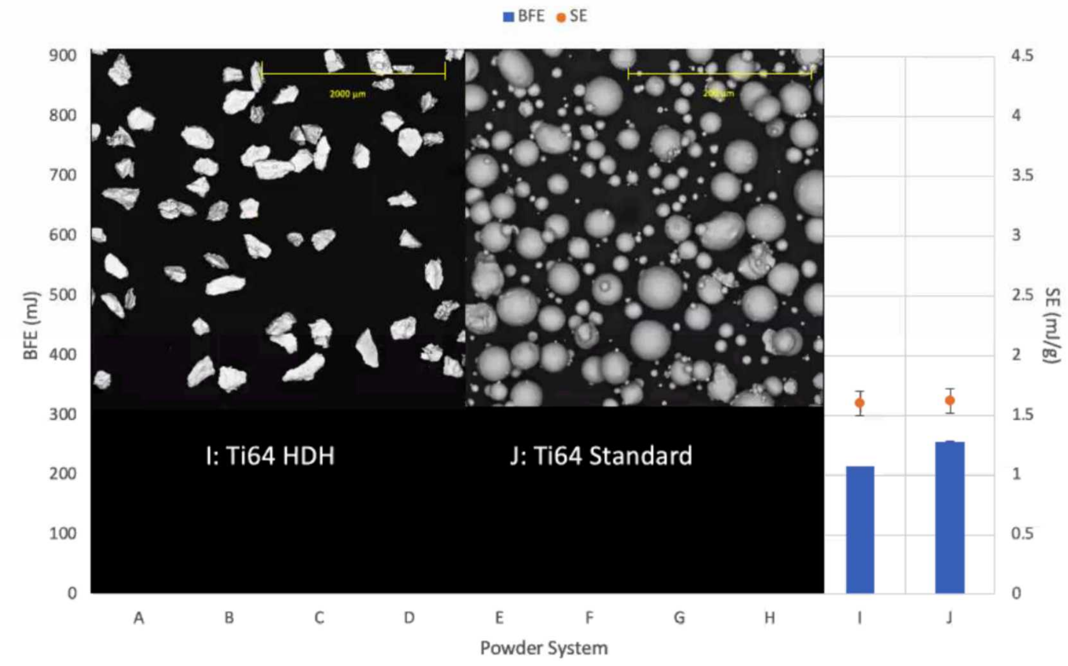
[1] Hebert, R. J. (2016). "Viewpoint: metallurgical aspects of powder bed metal additive manufacturing." Journal of Materials Science **51**(3): 1165-1175.
[2] I.E. Anderson, E.M.H. White, R. Dehoff, Feedstock powder processing research needs for additive manufacturing development, Current Opinion in Solid State and Materials Science 22(1) (2018) 8-15.
[3] Heiden, M. J., et al. (2019). "Evolution of 316L stainless steel feedstock due to laser powder bed fusion process." Additive Manufacturing **25**: 84-103.

Important Powder Characteristics to Track

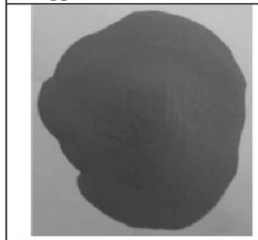
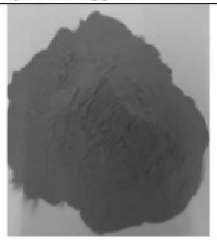
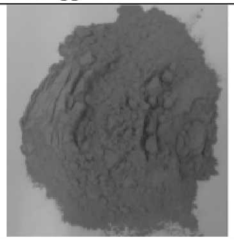
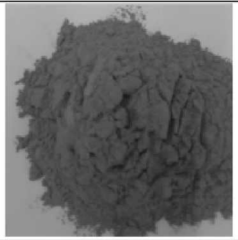
Tribocharging effects change with reuse [1]



- Triboelectric charging and charge density on particles tend to be higher for 316L virgin than reused, indicating less flowability



Surprising result: very different powder morphologies yield similar flow [4]

φ = 1 – very good flowability no agglomerations	φ = 2 – sufficient flowability Very loose agglomerations	φ = 3 – critical flowability loose agglomerations	φ = 4 – unsufficient flowability severe agglomerations
			

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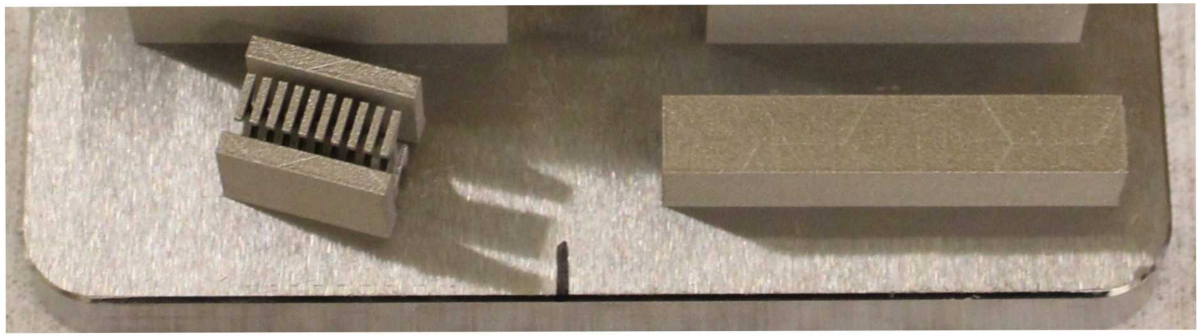
[1] Granutools. Flowability, bulk density and electrical charges - Additive manufacturing powders characterization. GranuTools Application Note. GranuTools.com (2017)
[2] Mercury Scientific Inc. AM Powder Testing. Flowability Application Bulletin 7.
[3] Spierings, A. B. Powder flowability characterization methodology for powder bed based metal additive manufacturing. Prog Addit Manuf. 1:9-20 (2016)
[4] Rollet, Tony, Carnegie Mellon University, Next Manufacturing Seminar

Coupling Powder & Mechanical Properties



High Throughput Tensile Sample

Charpy Test Coupon

Powder Sample

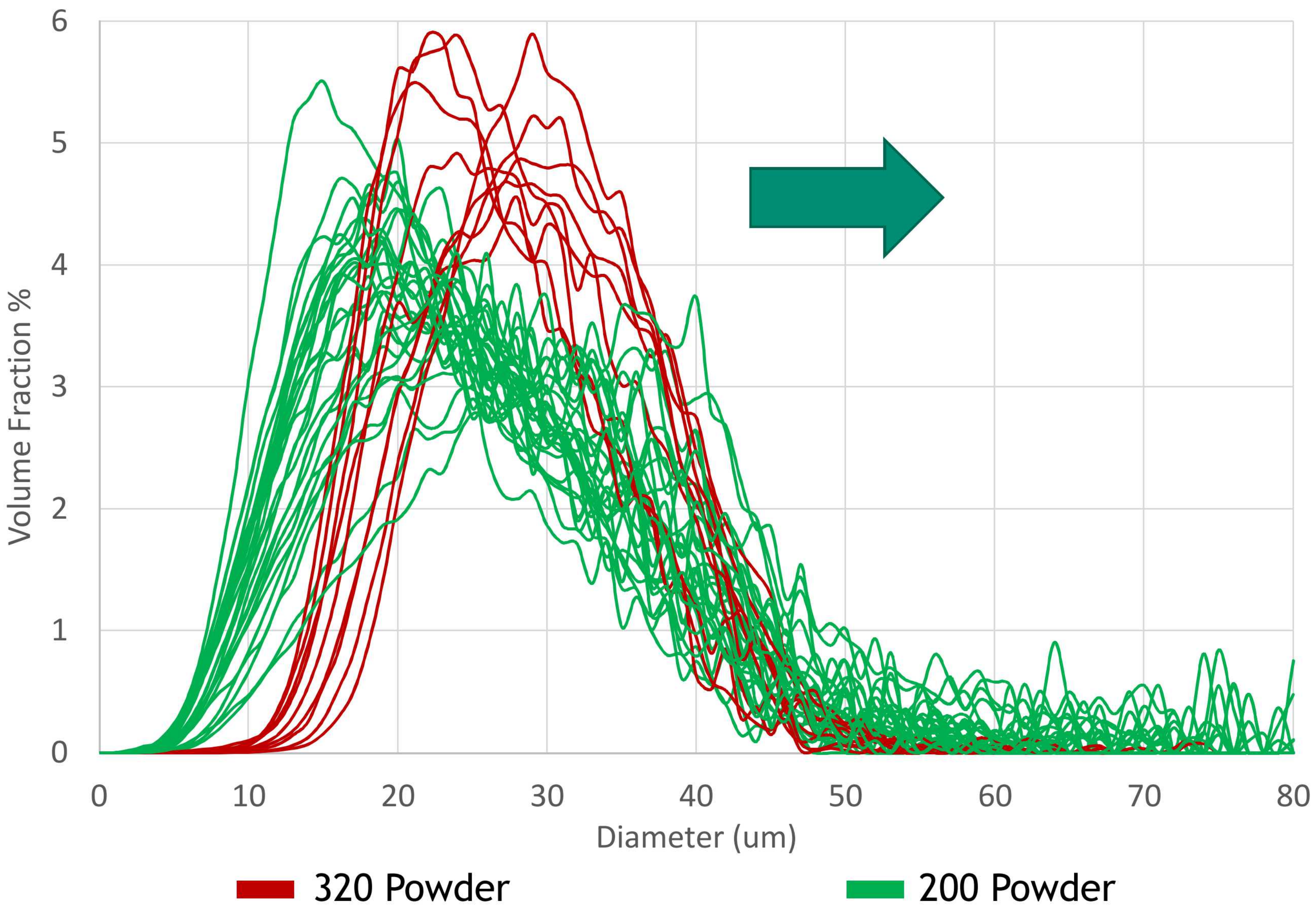
- Two witness coupons built on every build plate tracked since 10/03/18
- Powder lot sampled after sieving
- <u>Machine parameters set at:</u>
  - Power: 110 W
  - Mark Speed: 1400 mm/s
  - Hatch Spacing: 50 μm
  - Focus offset: 1.5 mm
  - Style: Hexagon
  - Layer Thickness: 30 μm
- <u>2 different types of 316L powder</u>
  - 200 powder
  - 320 Powder

1. Do part properties change with extensive powder reuse?
2. Which powder characteristics actually influence part properties?

<u>Properties tracked for each build:</u>

- Powder Morphology
  - Average Diameter
  - Size distribution: D10, D50, D90
  - Aspect Ratio
- Powder Bulk Chemistry
  - EDS
  - ICP-MS/OES + LECO
- Part Mechanical Properties
  - UTS
  - YS
  - % EL
  - Density
  - Fracture Toughness
  - Hardness

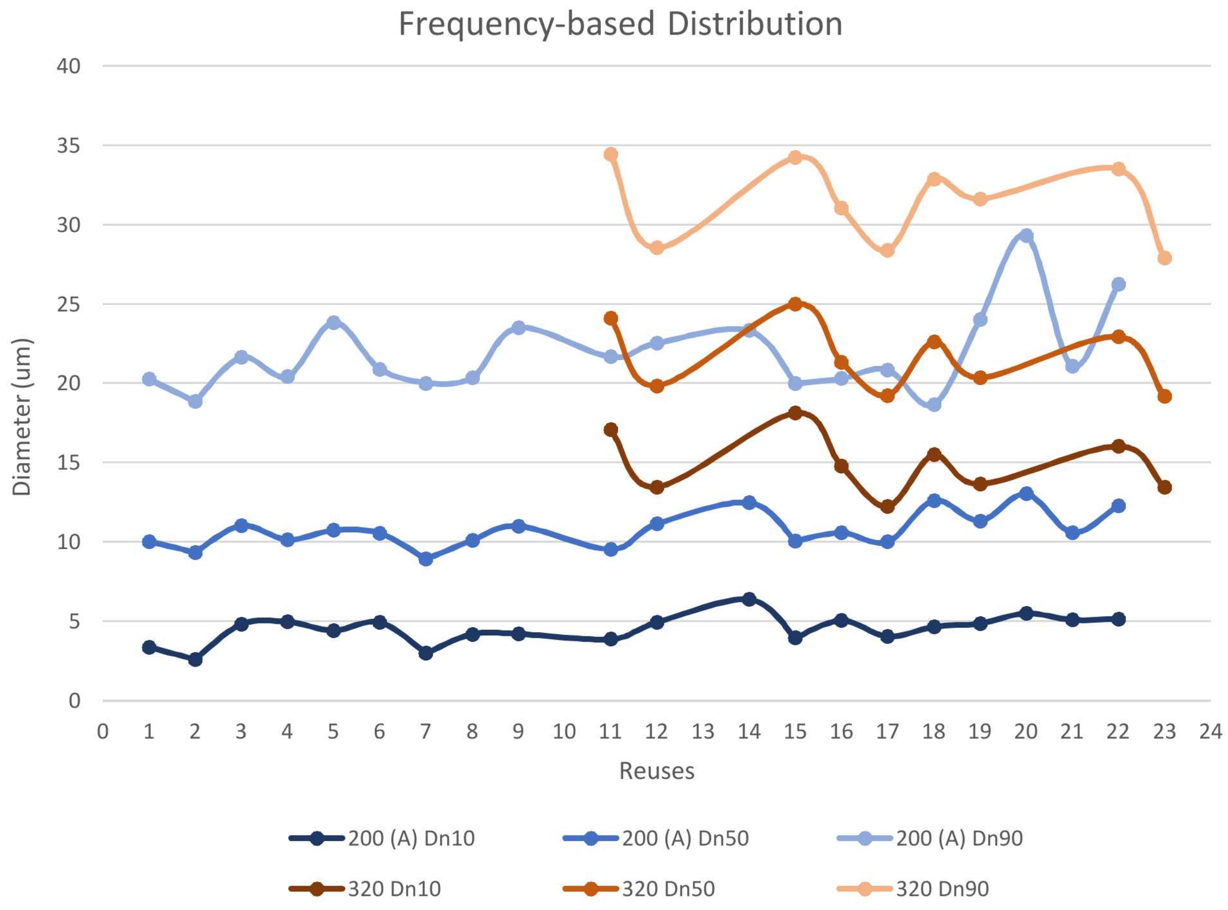
Reuse Effects on 316L for Varying Lots

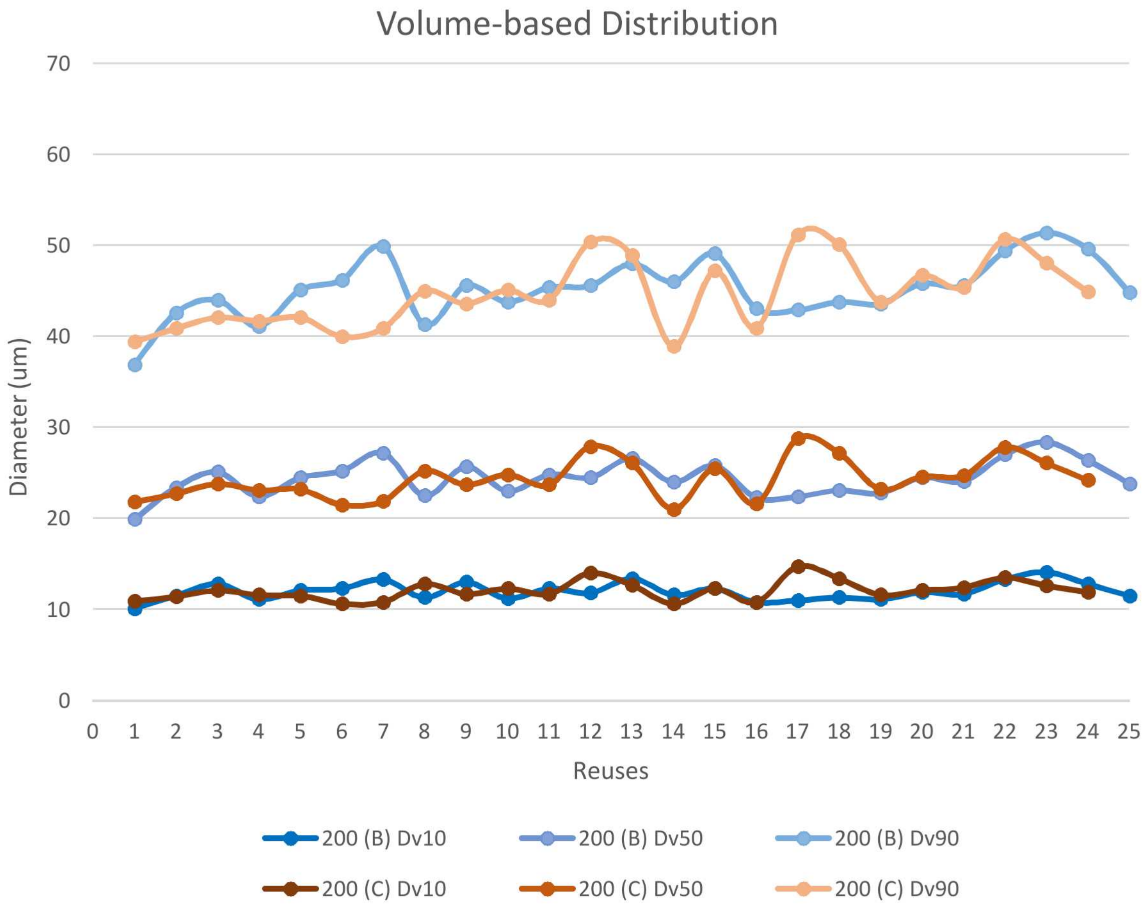


Lot	Average Diameter (μm)	Aspect Ratio
320	27.5	1.4
200-A	16.9	1.5
200-A Reused	16.7	1.5
200-B	14.4	1.4
200-C	11.6	1.5

Lot	D_n10	D_n50	D_n90
320	17.4	25.7	36.8
200-A	7.0	16.9	26.5
200- Reused	8.2	15.4	27.5
200-B	7.5	13.3	22.9
200-C	4.2	10.6	20.2

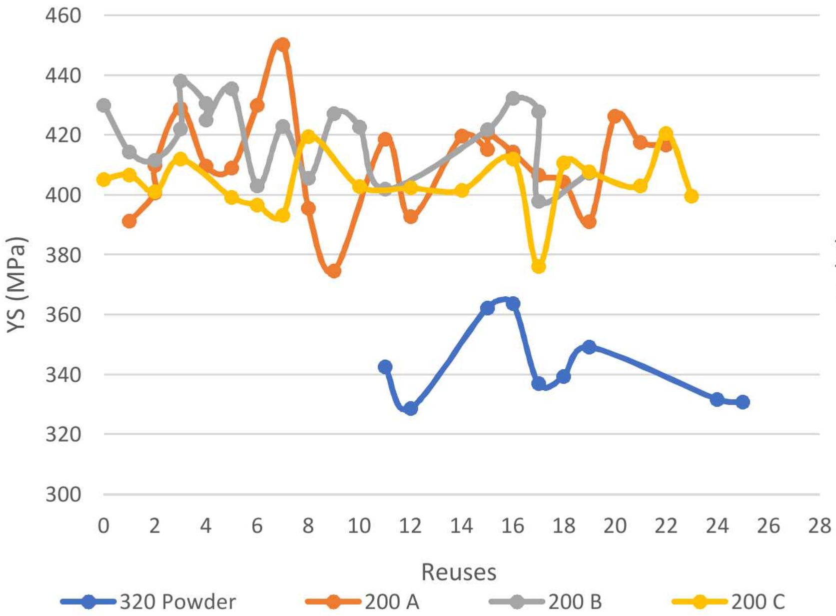
Particle Size Distribution Comparisons

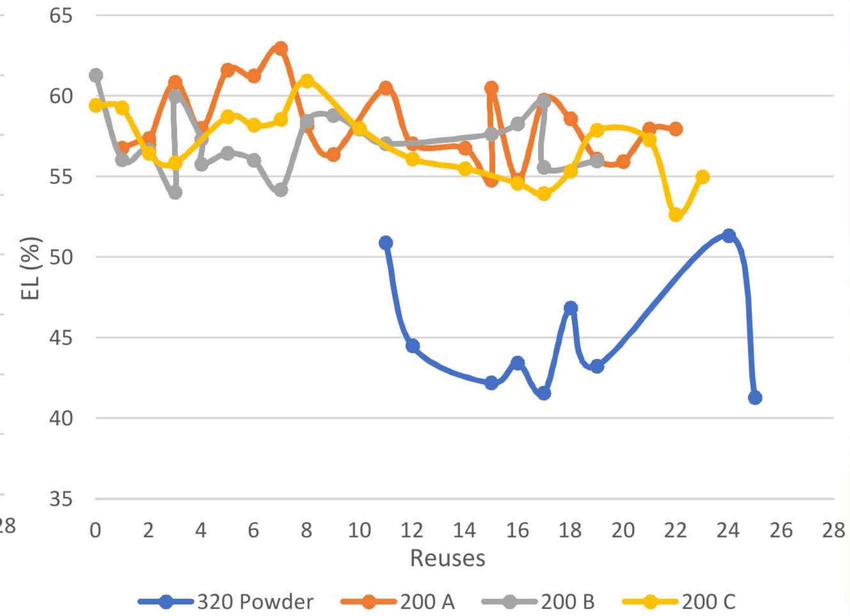




- Particle size distributions trend slightly upward to large diameters with reuse
- Some variation throughout – may be due to sampling

AM 316L mechanical properties insensitive to powder reuse

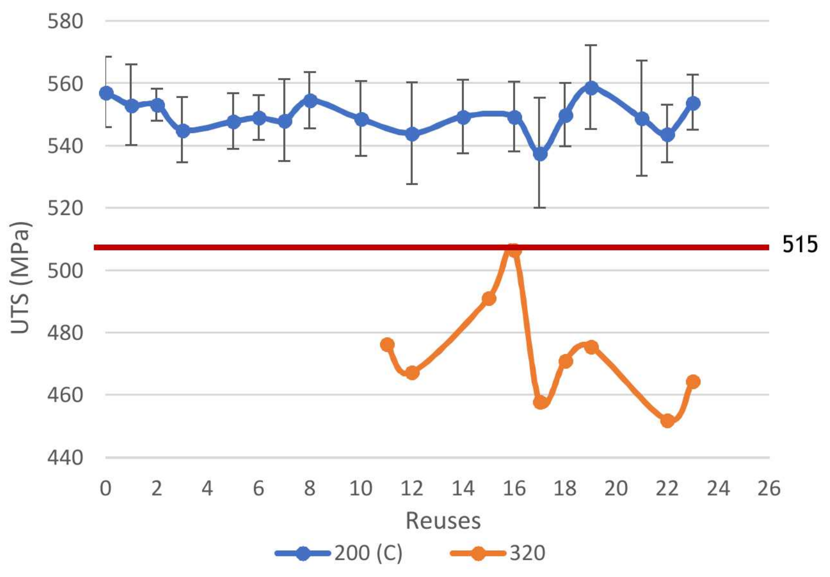


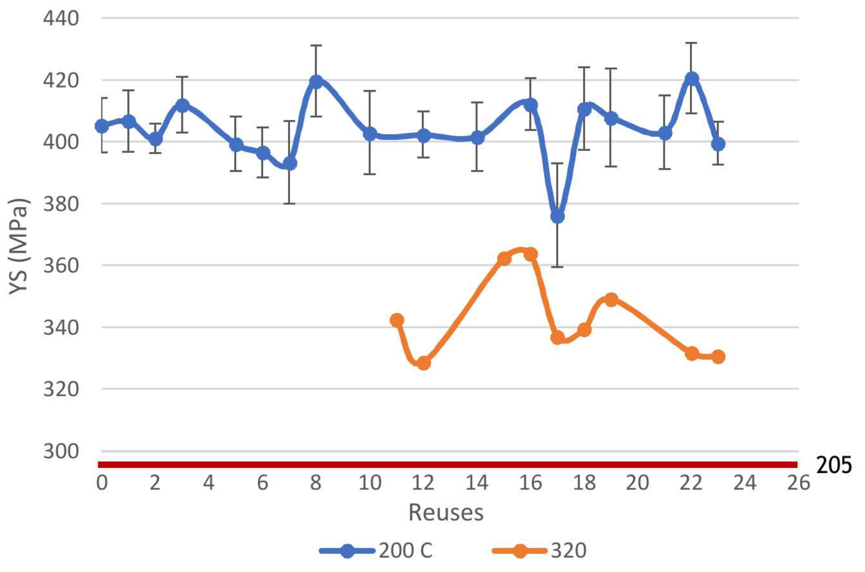


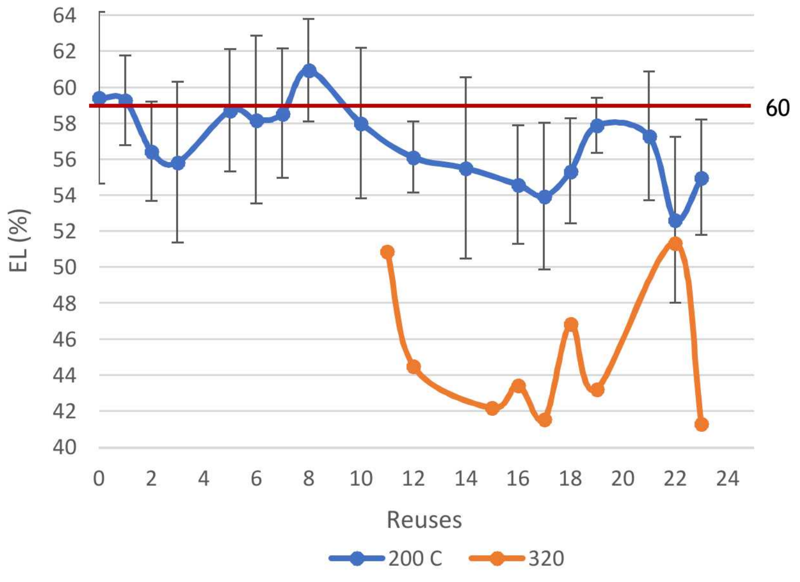
- PSD increases with reuse, but properties don't reflect that change
- Mechanical property variability may be primarily due to machine process variability, not powder

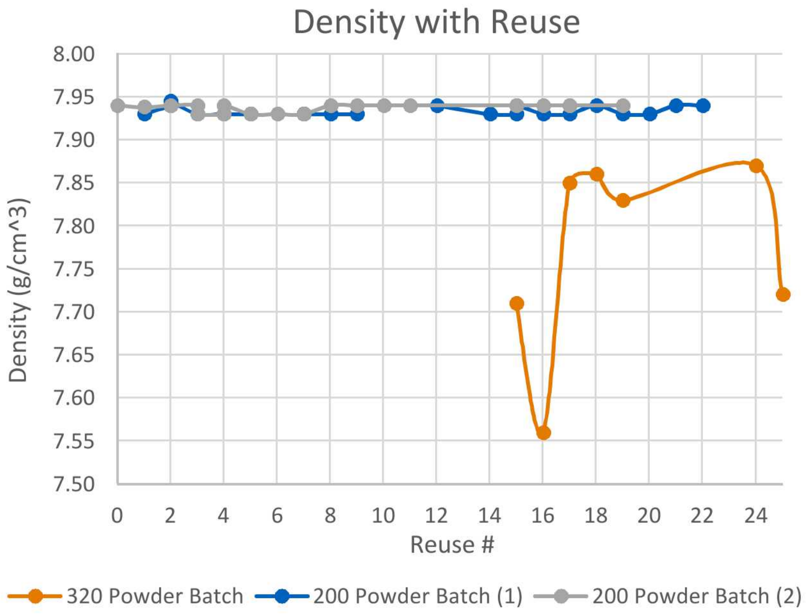
> - Density variation: ± 0.02 g/cm^3
> - Hardness variation: ± 6 HRB
> - UTS & YS variation: ± 70 MPa
> - Ductility variation: ± 16%
> - Fracture Toughness variation: ± 45 ft-lbs

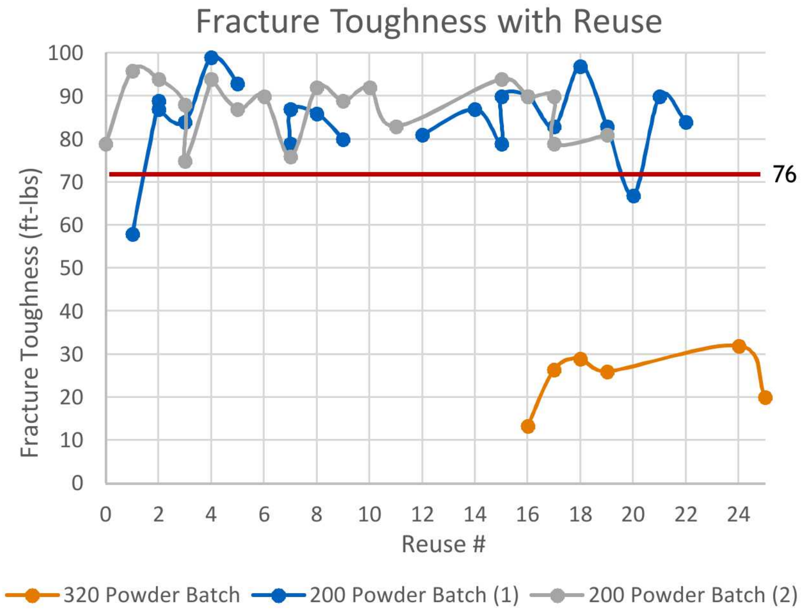
Mechanical properties fairly consistent across reuse cycles

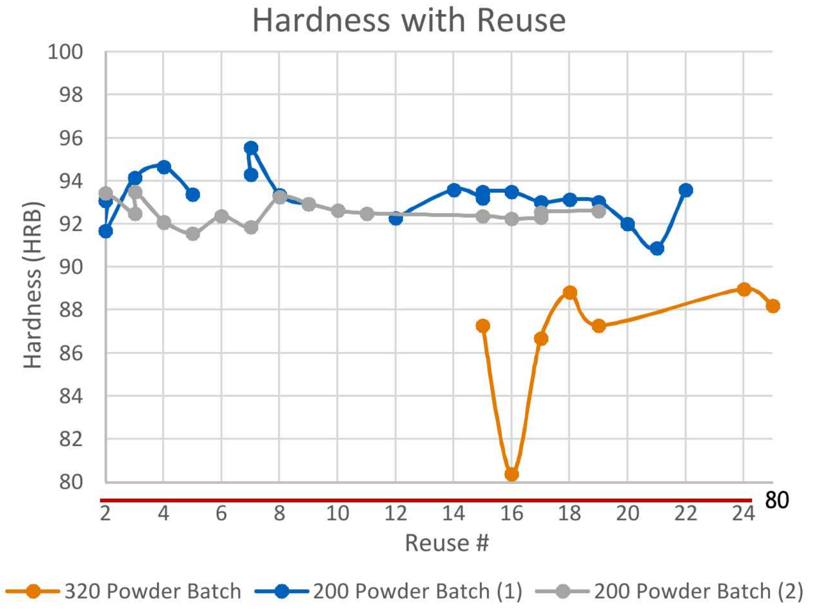




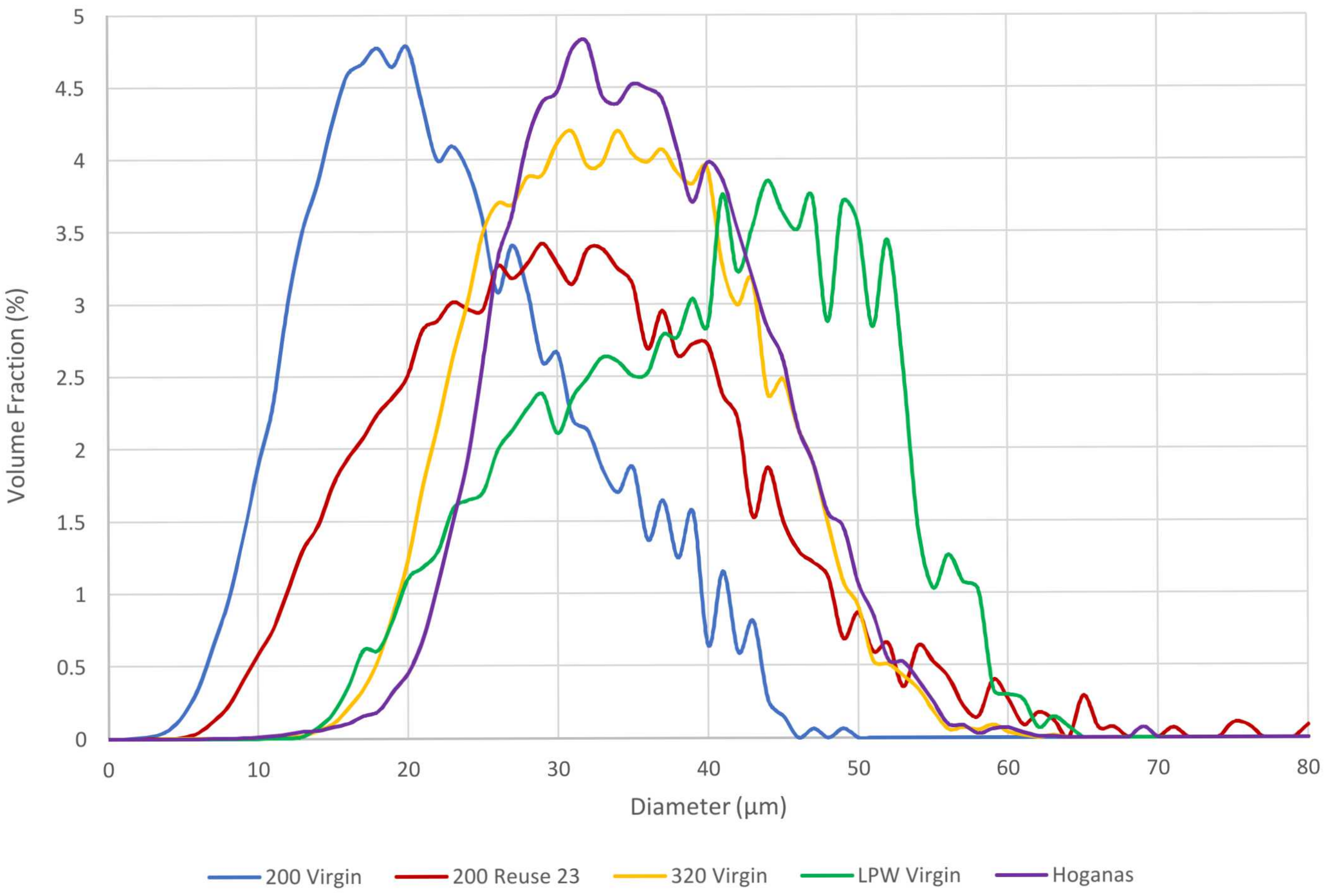


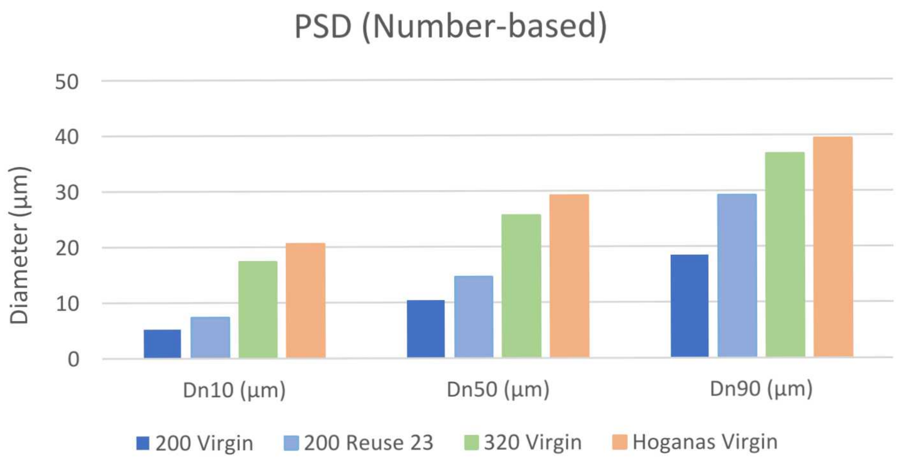


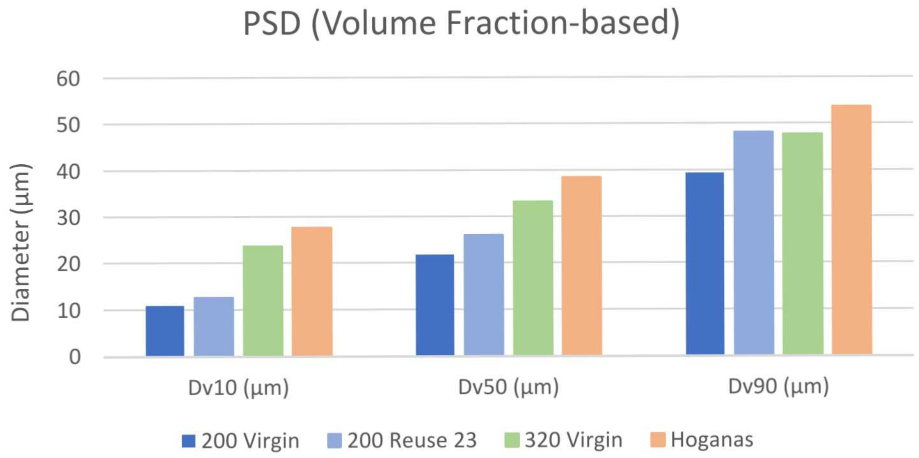


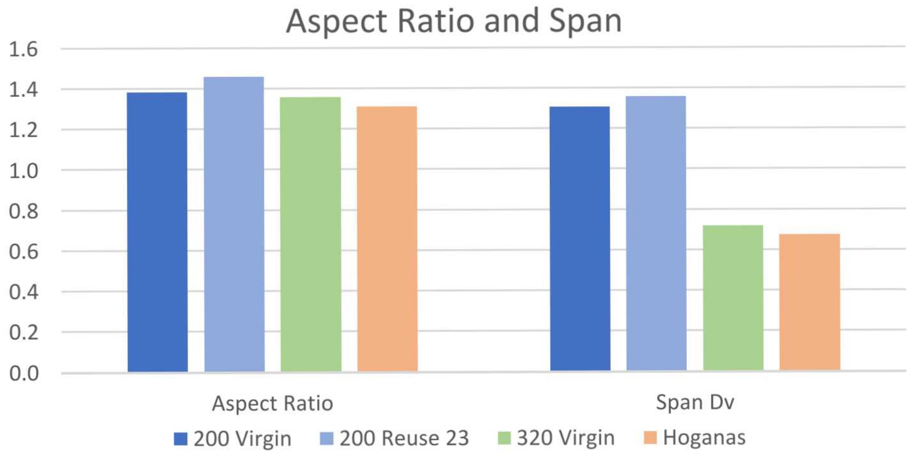


Comparing different starting powders



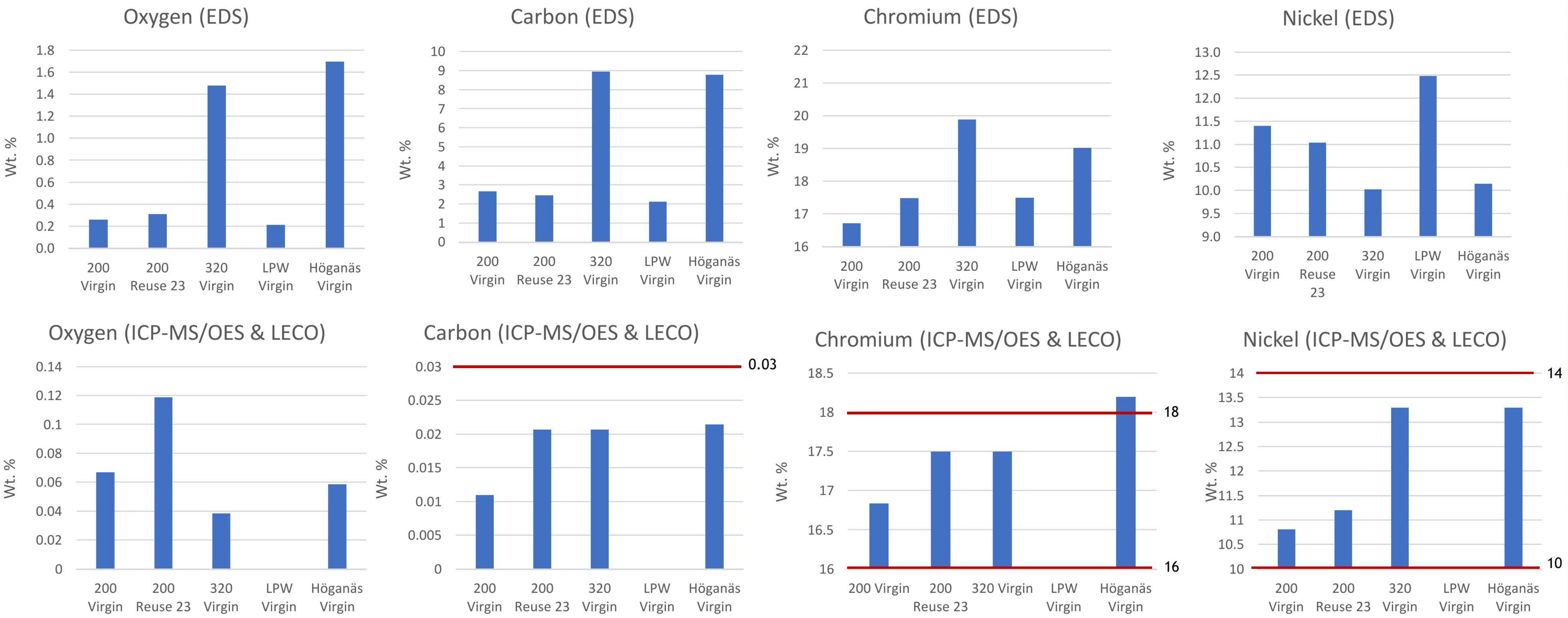






- Large variety of PSD distributions
- Virgin 320, LPW, and Höganäs powders fairly similar
- Reuse shifts distribution to the right and widens it

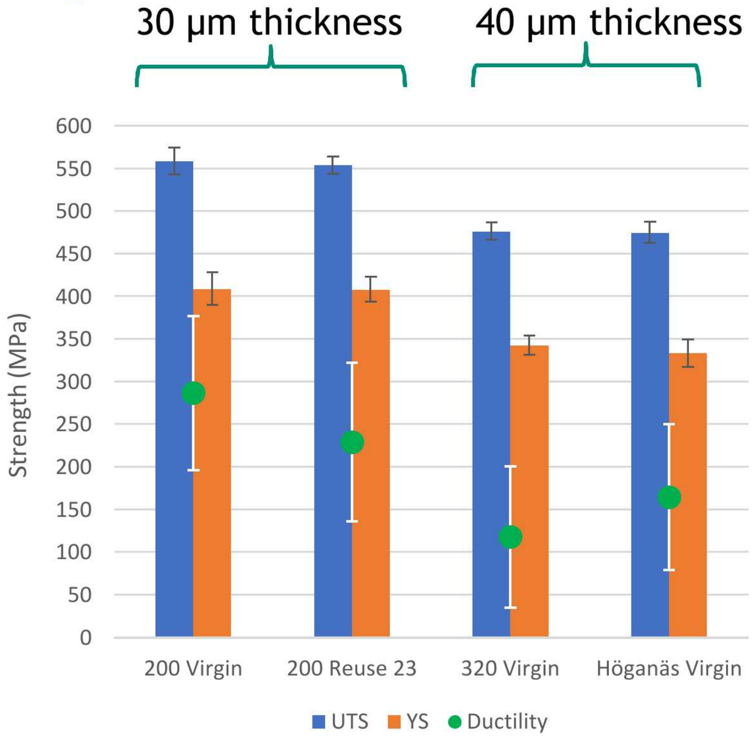
EDS not accurate enough for use in specifications

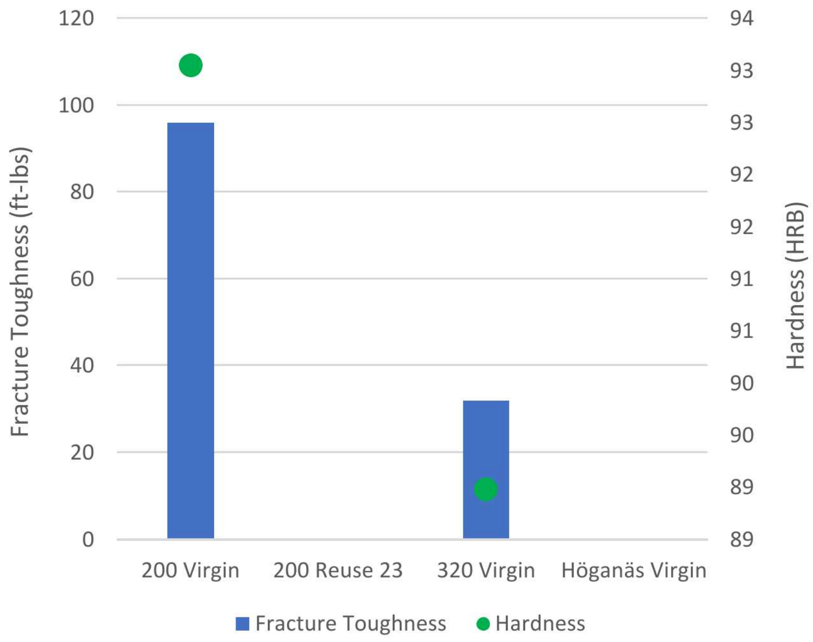


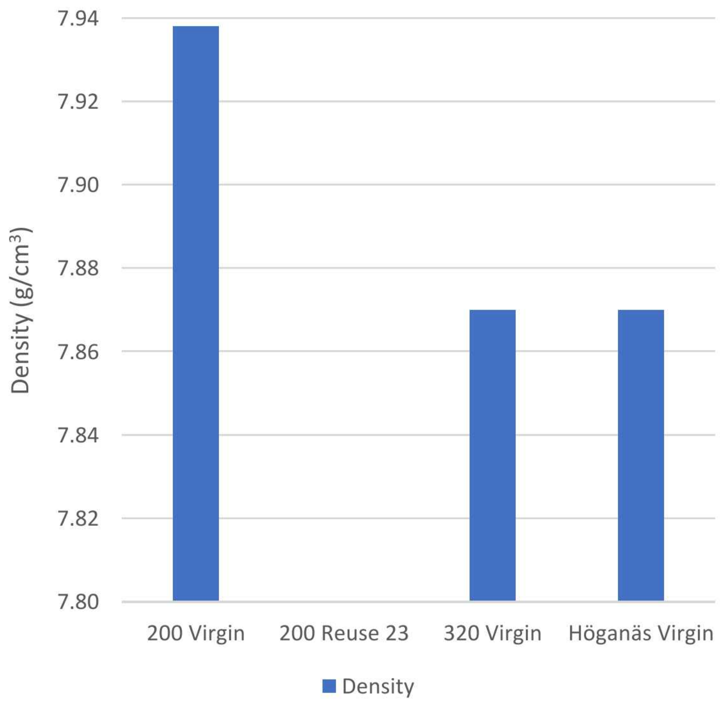
- Oxygen currently has no ASTM limit – could preliminarily set limit at 0.1 wt. %
- According to existing ASTM standard for 316L SS, Höganäs would be out of Cr tolerance

General Hypothesis: Powder with higher oxygen % produces weaker mechanical properties

Initial powder quality an indicator of resulting mechanical properties

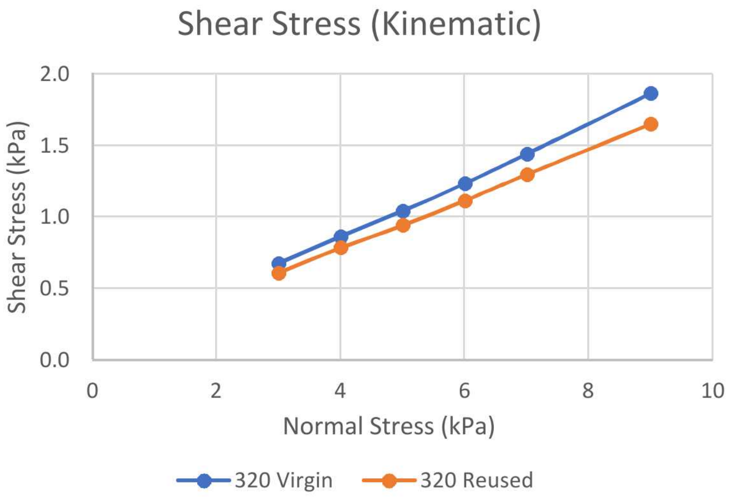


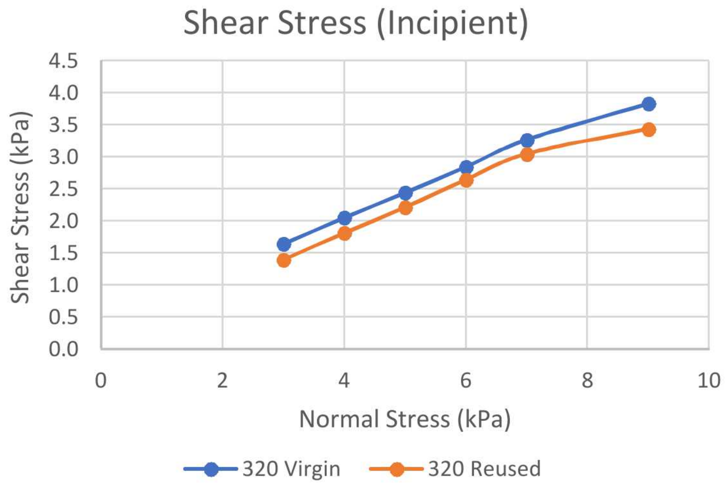


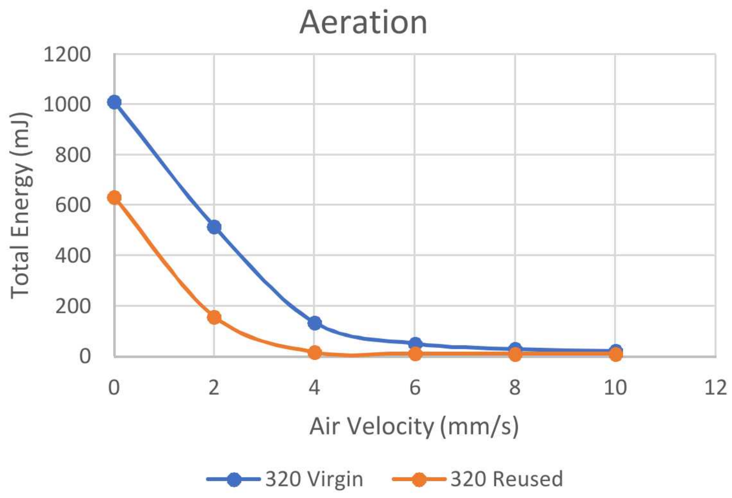


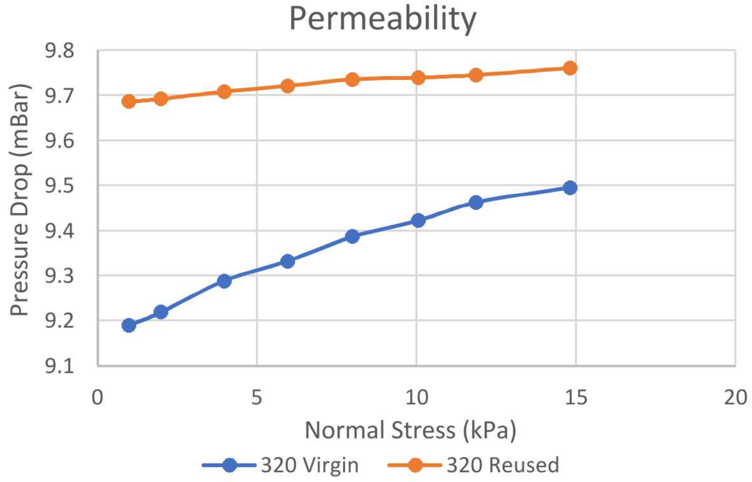
- "200 Reuse 23" has almost double O% without significant decrease in mechanical properties
  - This suggests PSD has greater influence on mechanical performance
- Initial PSD-machine layer thickness combo critical to optimize and "lock-in"
- Is there an optimal combo specific to each machine?
- Besides PSD, can any flowability metrics be used to quickly identify nonconforming powder?

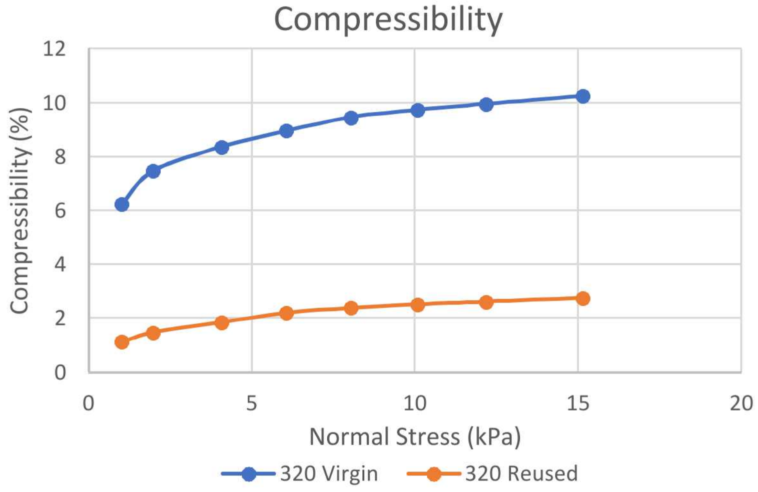
FT4 Rheometry of 320 powder reuse







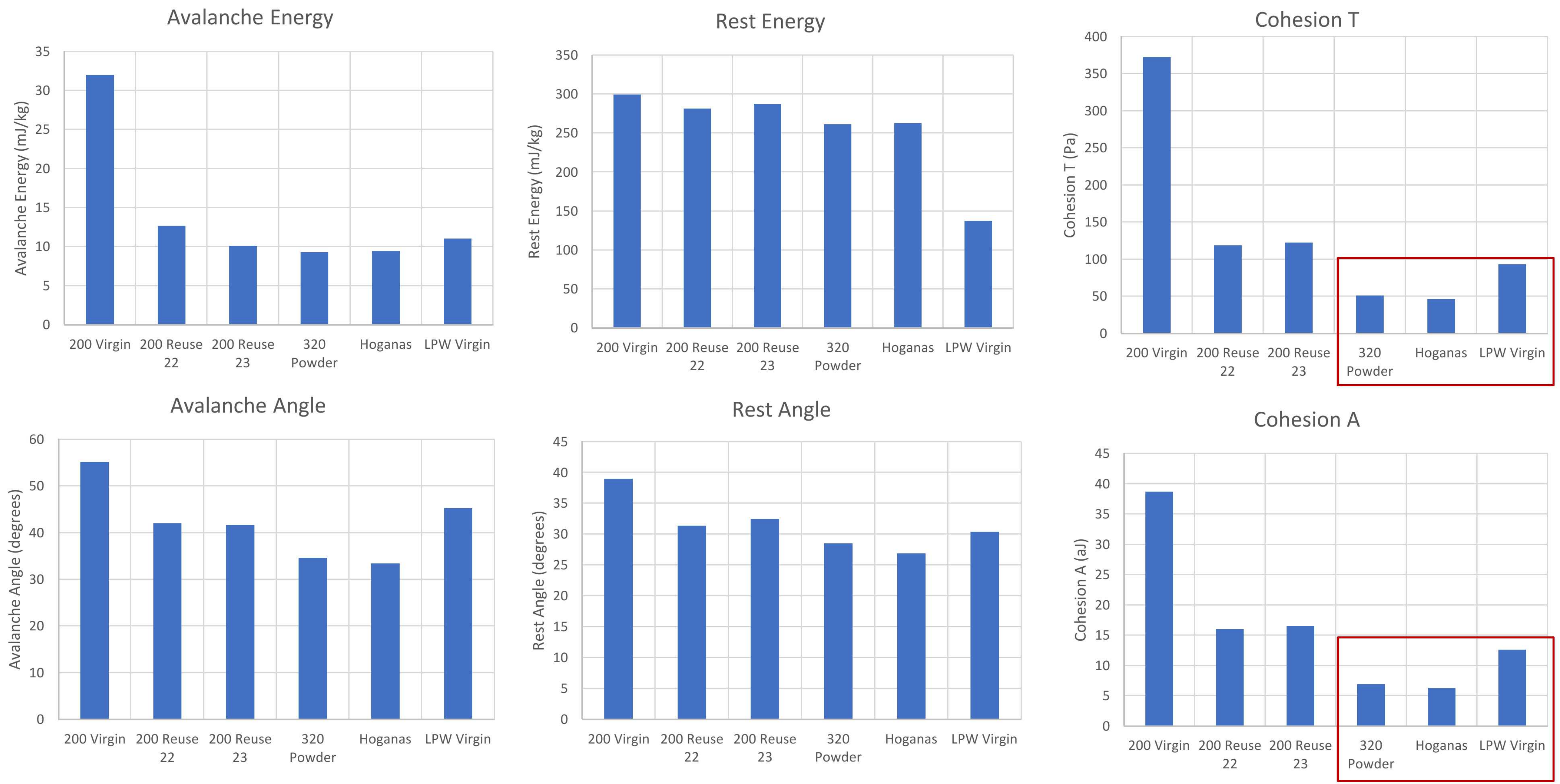




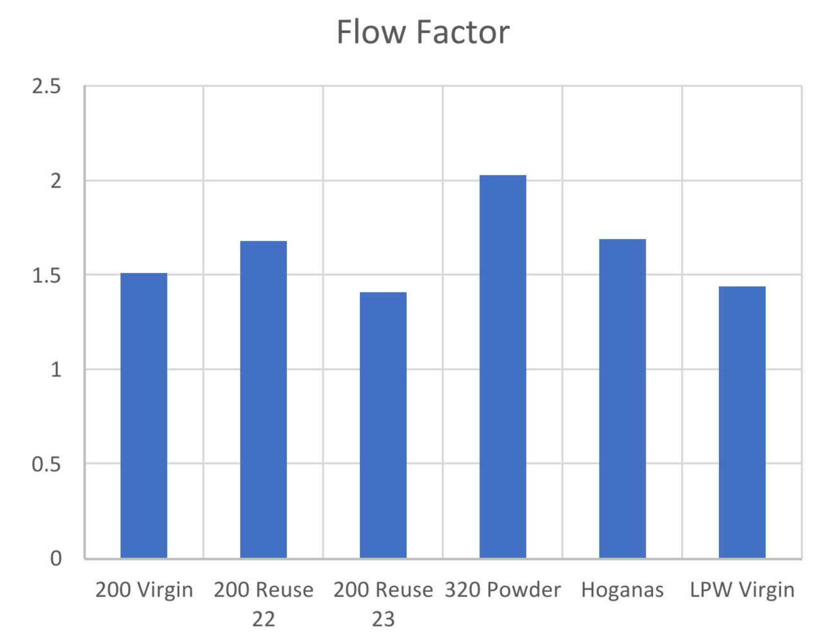
Metric	320 Virgin	320 Reuse 23
BFE (mJ)	991.76	1077.37
SI	0.97	1.00
FRI	1.04	1.13
SE	3.50	2.86
CBD	4.55	4.72
BDtap	4.84	4.66

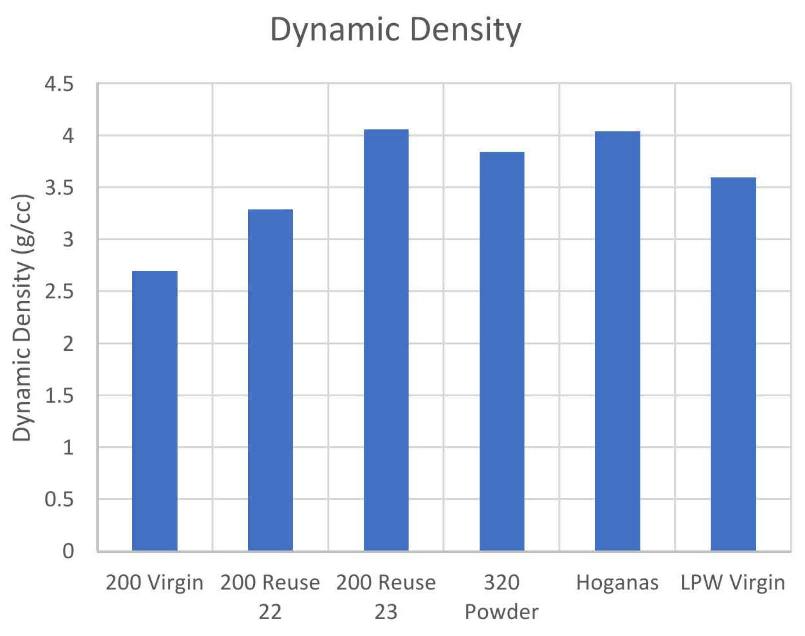
- Virgin powder: higher tap bulk density
- Reused powder: better packing ability, less entrapped air and better ability to release trapped air, less cohesive and lower tendency to agglomerate, lower mechanical interlocking, lower resistance/shear stress to flow, and more stable

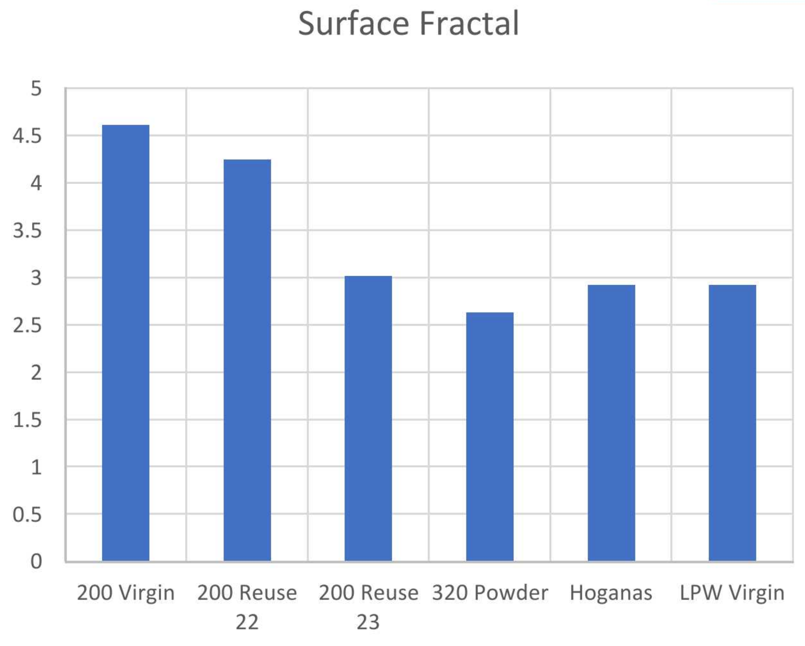
Flowability Trends (15 out of 130 flowability metrics show trends)

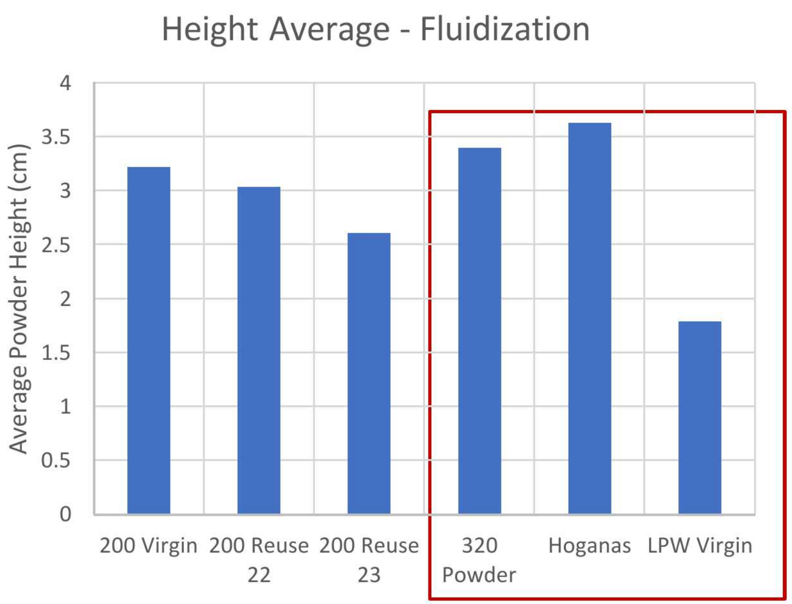


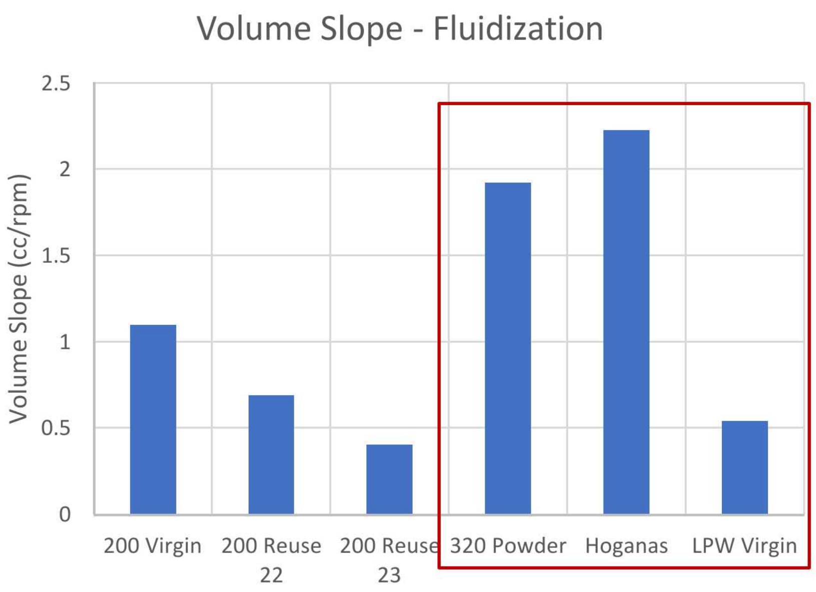
Flowability Trends (continued)



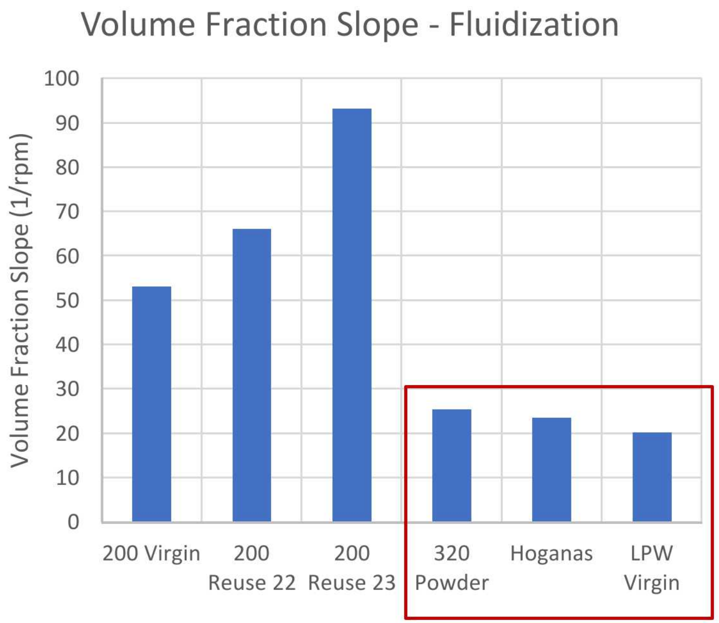


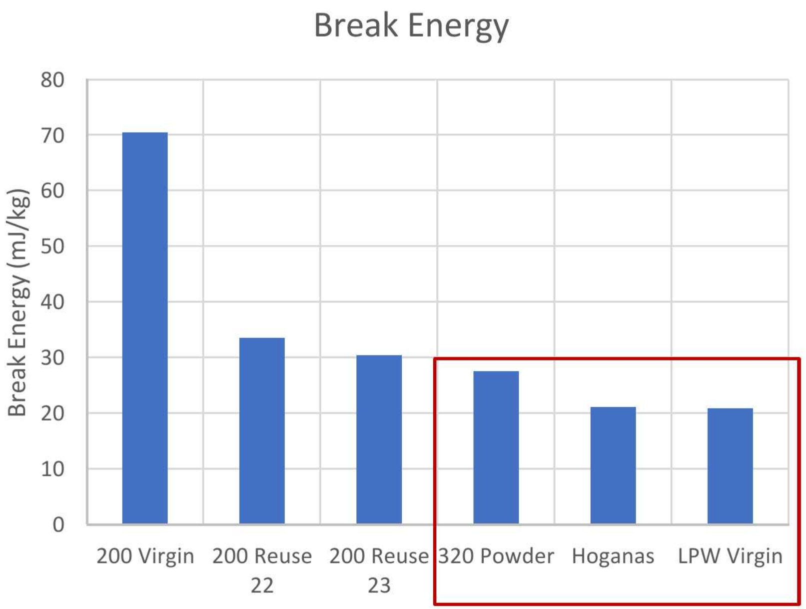


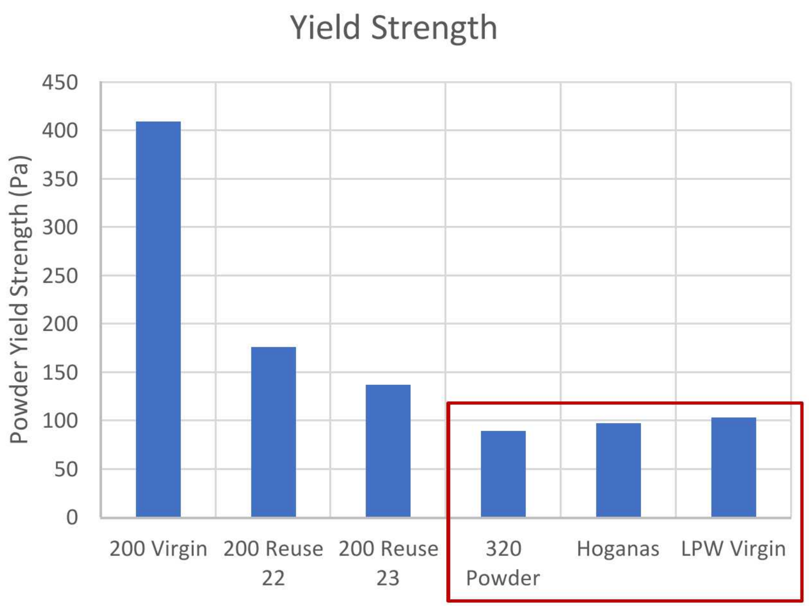




Flowability Trends (continued)

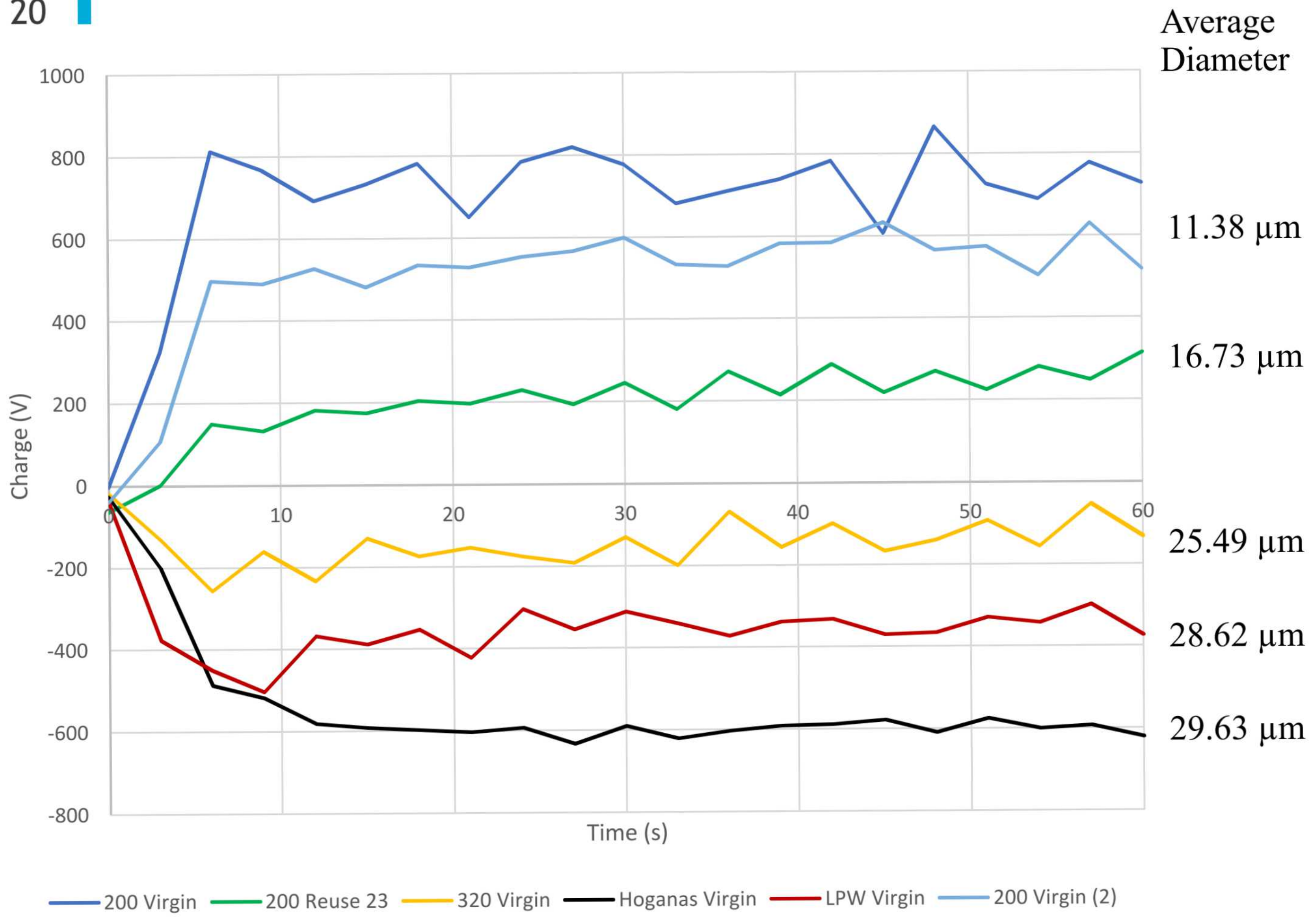


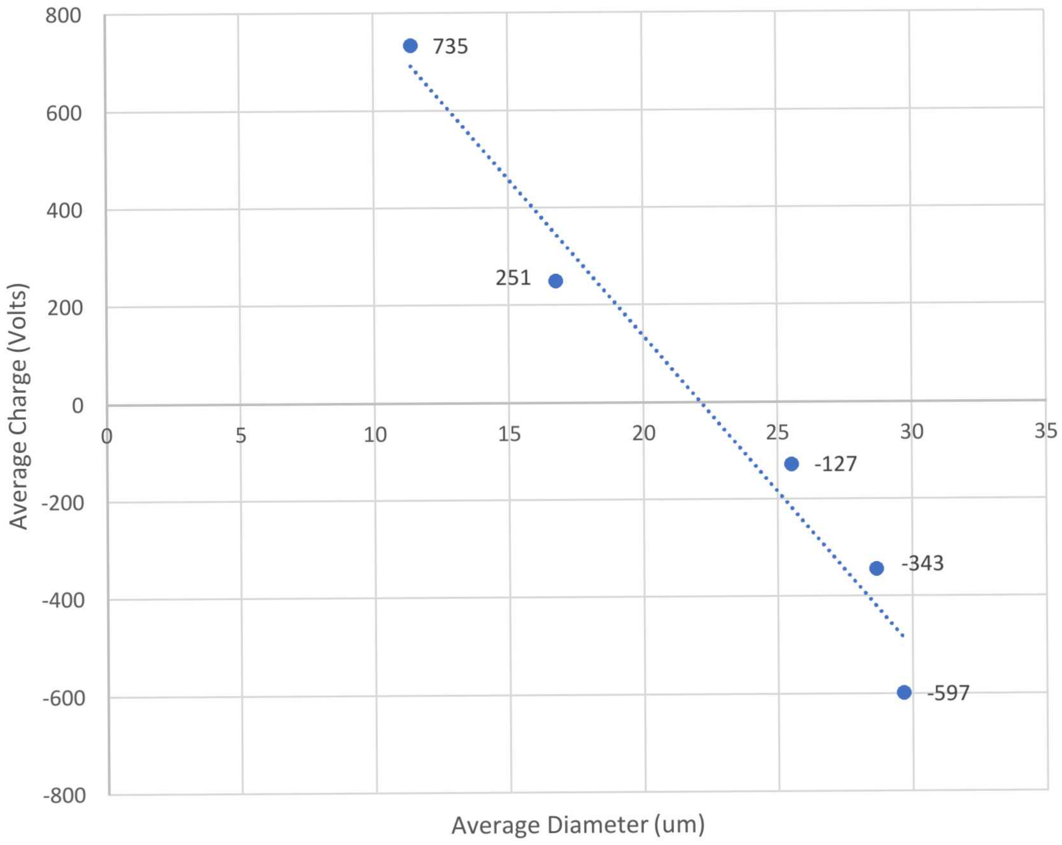




- Yield strength, break energy, & volume fraction slope show closest trends of differences between powder types according to PSD
- Cohesion A & T, height average, volume slope, & volume memory show slight trends between powder types

Charge Density Variation with Powder Lot

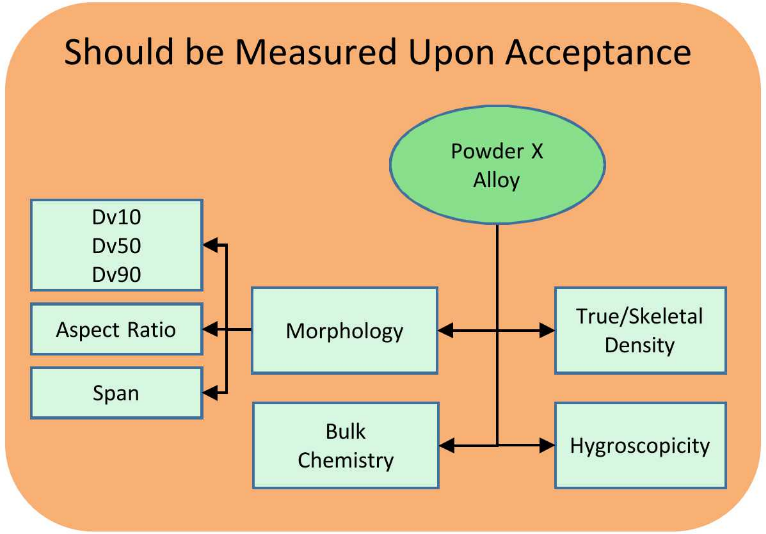


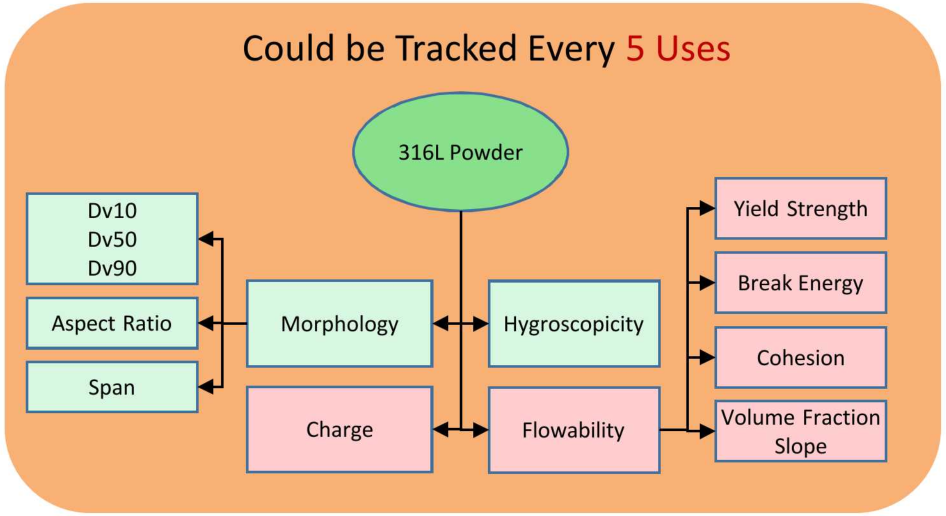


- Powders with largest avg. diameters require larger layer thickness, reducing mechanical properties at same parameters
- Larger powder sizes switch their plateaus to a negative charge
  - Reason unknown, size range unknown, effects on resulting parts unknown

Final Remarks: One Specification per Alloy/AM-machine Combination

- Type of powder used depends on part requirements & vendor supply
- Essential to tailor machine parameters according to powder size distribution
- Starting powder PSD is prime indicator of mechanical properties threshold
- A variety of powder characteristics change with 316L reuse
- 316L mechanical properties insensitive to powder reuse for ProX200, as long as D_v50 doesn't go above machine layer thickness
- Property variability primarily due to AM machine & location variability
- Different metal alloys will require more stringent limits on required characteristics (moisture and oxygen content for Ti and Al alloys)
- Each machine type handles and interacts with powder differently (blade vs roller, L-PBF vs EBM) so powder characteristics may need to be tied to specific machines to assure reliability
- Coupon density a reasonable indicator of any issues in powder quality





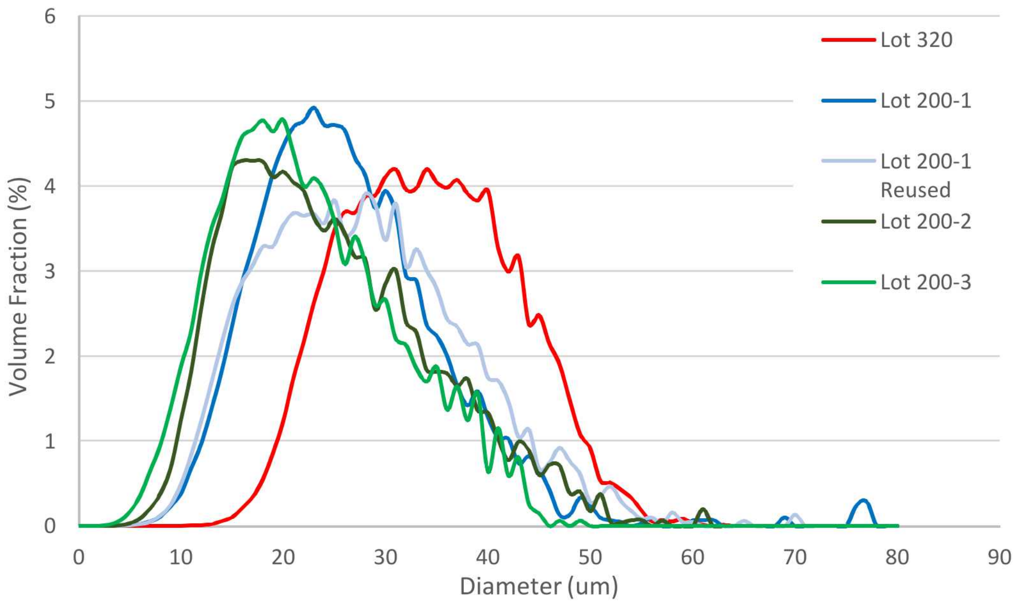
Appendix

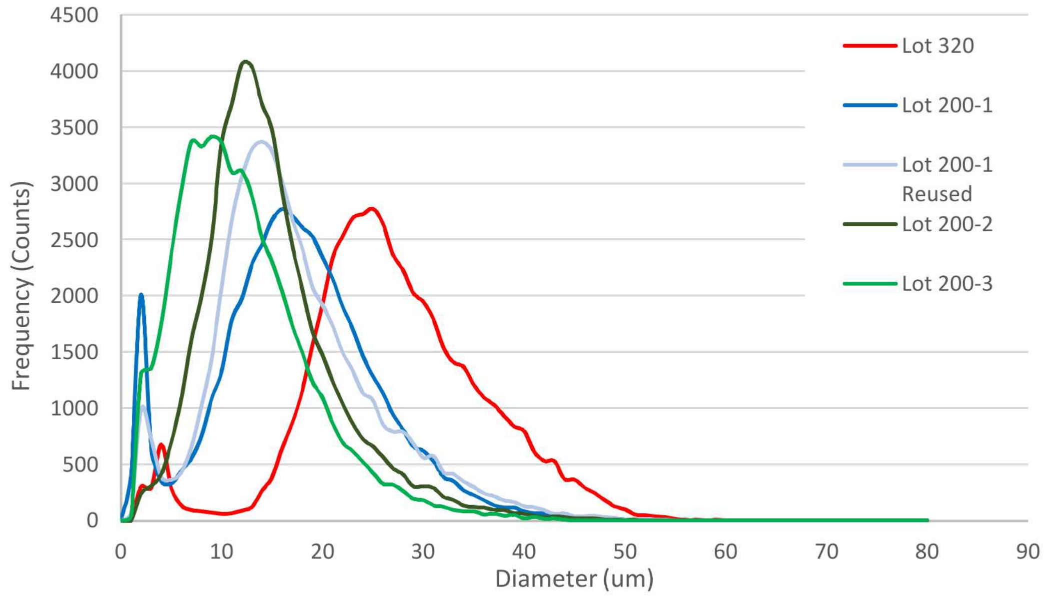
Flowability Criteria

	Criterion	FT4 indices
Powder bed density	Good packing ability	↑Conditioned bulk density (ρ_c)
		↓Compressibility (CI)
	Low entrapped air	↑Conditioned bulk density (ρ_c)
		↓Compressibility (CI)
	Good ability to release entrapped air	↑Permeability (↓PD)
Powder bed uniformity	Low tendency to agglomerate	↓Aeration energy (AE)
	Low mechanical interlocking	↓Specific energy (SE)
		↓Cohesion coefficient (c)
Minimal disturbances	Low resistance to flow	↓Specific energy (SE)
		↓Basic flow energy (BFE)

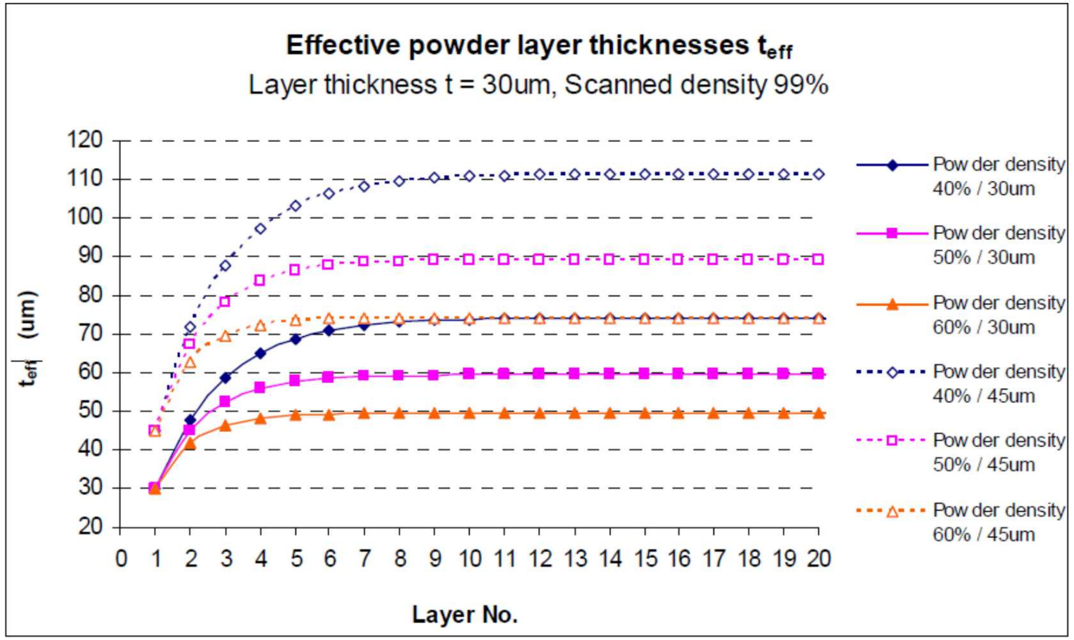
↑: index to maximize, ↓: index to minimize

Particle Size Distribution Comparisons





Influence of Powder Size on Laser Interactions



Suggestion:

$$10\ \mu\text{m} < D_{50} < 20\ \mu\text{m}$$

<u>General Feedstock Suggestions</u>

$$\frac{t_{eff}}{D_{90}} \approx 1.5$$

- The real (t_{eff}) powder layer thickness should be at least 50% higher than the diameter of 90% of powder particles

- Hoganas: 1.25 320 powder: 1.66 200 powder: 2.5

$$\textbf{Suggestion:}\quad 2 < \frac{t_{eff}}{D_{90}}$$

$$\frac{D_{90}}{D_{10}} \approx 5$$

- Suggests there needs to be a sufficient amount of fine particles in the distribution to fill voids between coarser particles

- Hoganas: 1.91 320 powder: 2.10 200 powder: 4.24

$$\textbf{Suggestion:}\quad 4 < \frac{D_{90}}{D_{10}} < 5$$

Spierings, A. B. (2011). "Influence of the particle size distribution on surface quality and mechanical properties in AM steel parts." Rapid Prototyping Journal **17**(3): 195-202.
Spierings, A. B. (2009). "Comparison of density of stainless steel 316L parts produced with selective laser melting using different powder grades." SFF Symposium 2009, 342-353